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Activated carbon production by co-carbonization of feathers using water-soluble phenolic resin under controlled graphitization

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ABSTRACT

We attempted to use feathers for the production of activated carbon (AC). A water-soluble resol-type phenolic resin was hybridized to prevent the liquefaction of the feathers and to control the graphitization degree of charcoal. The hybridization could effectively increase the yield of charcoal over 30% and maintained the graphitization degree at approximately 0.1, suitable for the production of AC. The Brunauer–Emmett–Teller (BET) surface area and the iodine-adsorption capacity of hybrid charcoal-based AC were 706 m^2/g and 550 mg/g, respectively, which were 1.7-1.8 times greater than those of the feather-based AC at a similar activation level.

1. Introduction

Pitch and coke, the heavy fractions of petroleum and coal, respectively, are the two major starting materials for obtaining various carbons, such as carbon blacks, carbon fibers, and activated carbons (ACs). Their carbonization behavior and the properties of the resulting carbons have been extensively studied. However, such carbon resources are not sustainable because of their limited reserves and the rising price of oil. Instead of fossil fuels, we have proposed alternative starting materials, such as coffee lees [1], triacetylcellulose waste [2], and oak woods [3], to produce AC. As for the cost of the resources, bottom ash [4] and waste rubber tires [5] are attractive, and adsorbents applicable to the wastewater treatments have been developed.

To establish the mass production of AC from biomass, it is important to secure the resources from the viewpoint of supply chain costs. In the case of woody biomass, it is sometimes difficult to secure a healthy supply of waste wood chips from lumber mill factories because normally they are scattered in the mountain areas, which raises the costs for the development of transport networks in terms of the effective agglomeration of waste wood chips. In contrast, poultry industries have already established their transport networks. Therefore, we can expect a healthy supply of waste feathers, preventing the extra costs for transportation and agglomeration; for example, the American poultry industry deals with more than 4 billion lb of chicken feathers per year [6].

As for the application technologies for the waste feathers, the anaerobic (oxygen-free) digestion process producing biogas energy and manure for agricultural use has been developed and is used worldwide [6]. To develop more functional materials from waste feathers, several trials have been conducted, e.g., self-organizing oligopeptides for synthesizing new biopolymer [7], bioplastic [8], and regenerated fiber production [9]. However, these material recycling processes require the purification of waste feathers. In the case of AC, the contamination of chicken blood in the feather waste will not affect the quality of AC because both chicken feathers and chicken blood can be similarly converted into carbon. In addition, concerning the structure of feathers, it can be assumed as an assembly of short hollow fibers. Thereby, feathers have excellent strength-to-weight ratios as well as large surface areas. For the production of AC, the hollow structure of feathers would be advantageous if its structural features could be retained during the carbonization process. Concerning the hollow structure, carbon nanotubes (CNTs) with hollow and layered nano-sized structures

are attractive to separate or remove the hazardous pollutants by functionalizing the hollow structure with acids or oxidizing agents [10,11]. However, it seems difficult to use CNTs for the daily wastewater system considering its running costs. Instead, if the feather can be carbonized while retaining its hollow structure, the carbon obtained will be functionalized to some extent, similarly as in the case of CNTs via chemical treatments, although their hollow structure is in a micrometer scale.

In this study, we have focused on the fundamental carbonization behavior of feathers. To prevent the feathers from undergoing liquefaction during the carbonization process and to control the graphitization properties of keratin molecules, we used a water-soluble phenolic resin as the feather modifier and investigated the potential of waste feathers as a starting material for the production of AC.

2. Experimental

2.1. Materials

Feathers were obtained from bedclothes waste that underwent dry-cleaning several times. Water-soluble phenolic resin (resol type IG-1002, nonvolatile: 50%) was supplied by DIC Co. (Tokyo, Japan). Other chemicals used in this study were all laboratory-grade from Wako Pure Chemical Industries, Ltd. (Osaka, Japan).

2.2. Carbonization and activation at the laboratory-scale

A newly designed electric furnace (TL1, Carbo-tec Co. Kyoto) with an internal volume of approximately 81 L was used. For carbonization, a 125–200 g sample was packed at the bottom of a reaction vessel (with an internal volume of ca. 10 L and diameter of ca. 20 cm) by placing a stainless drop-lid and filling the gap between the edge of the lid and the wall of the vessel with ceramic fibers. Then, the sample was heated to 800 °C at a heating rate of 3.4 °C/min, kept at 800 °C for 60 min, and left alone to cool to room temperature. For the production of the hybrid charcoal from feathers and phenolic resin, a certain amount of feathers was pretreated via dipping in an aqueous solution of the water-soluble phenolic resin, followed by drying at 70 °C for 60 min. In the case of the water-soluble phenolic resin alone, pre-heating was sufficient to produce a cast film at the bottom of the reaction vessel. The charcoal samples obtained are listed in Table 1 with the respective codes used throughout the text. The mass ratio in Table 1 was defined as the relative mass of the feathers versus the mass of the phenolic resin on a dry base.

To activate the charcoal, a rotary kiln (with an internal volume of ca. 0.75 L) was used. First, 30–60 g of charcoal was heated to 900 °C at a heating rate of 10 °C/min under N_2 gas flowing at 0.6 L/min; then, water vapor was added. The activation lasted for 5 min.

2.3. Heat treatment at 3000 °C

Samples heat-treated at 800 °C were further heated from room temperature to 3000 °C in 140 min, kept at 3000 °C for 30 min, and cooled to room temperature using the electric furnace (SCC-U-120/203/135, Kurata Giken Co., Konan Shiga, Japan) under argon gas flow.

Table 1
Sample codes of the starting materials.

Sample code	Starting material
F4Ph1	Feather waste (F) and phenolic resins (Ph), mass ratio = 4:1
F1Ph1	F and Ph, mass ratio = 1:1
TRY	L-Tryptophan
CEL	Cellulose (reed grass)
ACE	Acenaphthylene
Pr1S4	L-Proline (Pr) and Shellac (S), mass ratio = 1:4

2.4. Thermogravimetric analysis/differential thermal analysis (TG/DTA) measurement

TG/DTA was performed using an analyzer (WS-002, Bruker AXS K.K., Yokohama, Japan) on a 10 mg sample at a heating rate of 20 °C/min, up to 1000 °C, under N_2 gas flow at 50 mL/min. The sample was prepared as follows. In the case of the feather/phenolic resin mixture, the milled feathers were dispersed into 50% phenolic resin aqueous solution and then dried at 60 °C for 60 min. The obtained film was milled, and 10 mg was sampled. As for the phenolic resin, the cast film obtained from its solution was used.

2.5. X-ray diffraction

Wide-angle X-ray diffraction (WAXD) was performed on isotropic samples, which were obtained by grinding and mixing the carbons with high-purity silicon powder as the inner standard. The X-ray source was CuKα radiation.

The degree of graphitization (P1), defined as the probability for adjacent hexagonal carbon layers to have positional correlation in the graphite, was determined from the Fourier coefficients of both the 101 and 112 diffraction profiles [12,13], which were measured using step-scanning with a step of 0.01 and an accumulation time of 30 s. The average interlayer spacing was calculated using Bragg's equation from the peak diffraction angles of the carbons determined by referring to the diffraction of the silicon standard. The average interlayer spacings determined from the 002, 004, and 006 diffractions were denoted as $d_{002}(002)$, $d_{002}(004)$, and $d_{002}(006)$, respectively. The crystallite sizes parallel and perpendicular to the c-axis (Lc and La) were calculated from the full-width at half-maximum for the diffraction peaks of the carbons, which were corrected for the instrumental peak broadening by referring to the diffractions of the silicon standard. The size parameters determined from the 002, 004, 006, and 110 diffractions using Scherrer's equation were denoted as Lc(002), Lc(004), Lc(006), and La(110), respectively. The standard deviation of the distribution in the interlayer spacing (σ_c) and the true crystallite size (L_0) was determined using Hosemann's equation [14].

$$[1/Lc(00\ l)]^2 = [1/L_0]^2 + \pi^4 \sigma_c{}^4 l^4 / \left[16 d_{002}(004)^6\right] \tag{1}$$

where l is the order of diffraction.

2.6. Surface characterization

The N_2 gas adsorption capacity was determined using an adsorption measurement instrument (BELSORP-18plus, BEL Inc. Jpn, Toyonaka, Japan). The surface area (S) was determined using the Brunauer–Emmett–Teller (BET) plot [15]. The volume (V) was determined from the amount of N_2 adsorbed at a relative pressure of 0.98. The mean pore diameter (D) was calculated as $D = 4 V/S$ by assuming that the pores are uniform nonintersecting cylindrical capillaries.

The iodine adsorption capacity was determined with a titration method using 0.1 M sodium thiosulfate [16].

3. Results and discussion

3.1. Co-carbonization behavior of phenolic resin-modified feathers

For the production of carbonaceous adsorbents, the hollow structure of feathers would be advantageous if its structural features could be retained during the carbonization process. However, feathers tend to easily decompose hydrothermally and dissolve into water during hot pressurized water treatment over 180 °C [7]. Therefore, modifying the thermal properties of feathers from the viewpoint of thermosetting is

necessary. In this study, we have hybridized the water-soluble resol-type phenolic resin with feathers.

Fig. 1 shows the aspects of the untreated control feather waste and the hybrid charcoal obtained after the heat treatment at 800 °C. It is seen that the charcoal sample F1Ph1 hybridizing the phenolic resin with feathers at the mass ratio of 1:1 on a dry base (see the sample code in Table 1) could retain the shape of feather barbs almost completely even though the barbs were buried in the phenolic resin-based charcoal. In contrast, without hybridizing the phenolic resin, the feather barbs completely melted (see Fig. 1(d)).

The TG/DTA curves for the mixture of feathers and phenolic resin at a mass ratio of 1:1, i.e., the starting material of F1Ph1 charcoal, and the cast film prepared from phenolic resin are shown in Fig. 2. It is seen that the yields of charcoal at 800 °C were approximately 33% in both systems. However, each slope of the TG curve became quite different. From an industrial viewpoint, a system showing a mild thermal decomposition behavior is desirable to guarantee steady production. The water-soluble phenolic resin showing a slightly steeper slope around 800 °C had therefore better be used as the feather modifier rather than used alone.

The laboratory-scale carbonization treatment was conducted using the electric furnace. The yields of charcoal produced at 800 °C are listed in Table 2. The yield of charcoal was defined as the relative mass of charcoal against the mass of the starting sample. The feather-based charcoal exhibited a slightly smaller yield than the phenolic resin-based one, although both yields estimated by TG measurements lay within the same range at 800 °C. This may result from the partial combustion of feathers induced by air packed during carbonization sample
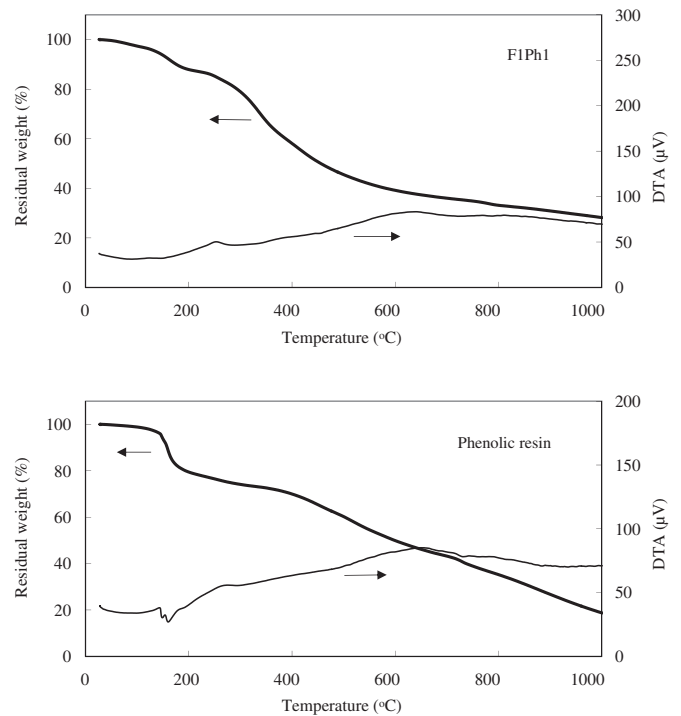

Fig. 2. TG/DTA curves: top, a mixture of feathers and the phenolic resin at a mass ratio of 1:1 (F1Ph1); and bottom, cast films obtained from the phenolic resin.
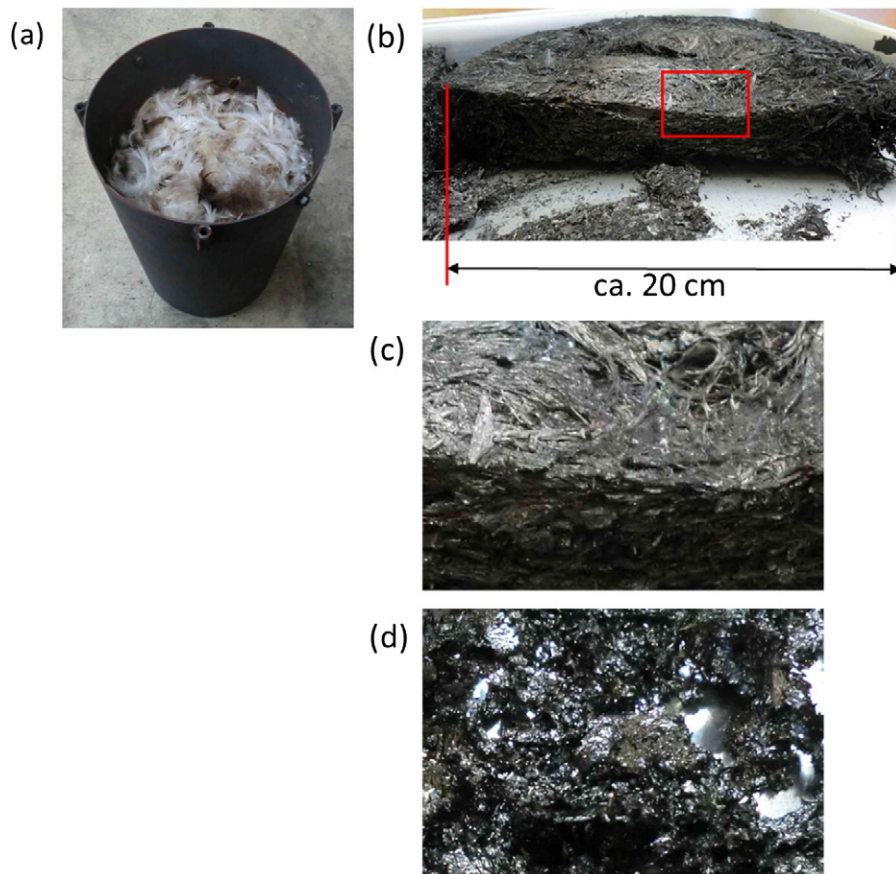

Fig. 1. Aspects of the starting material and obtained charcoals after heat treatment at 800 °C: a) starting feathers in the stainless reaction vessel, b) charcoal from a mixture of feathers and the phenolic resin at a mass ratio of 1:1 (F1Ph1), c) enlarged view of the portion surrounded by the square in Fig. 1b and d) charcoal of feathers.

Table 2
Yields of charcoal produced at 800 °C.

Sample code	F	Ph	F4Ph1	F1Ph1
Measured	15.6	25.1	20.0	30.5
Calculated	–	–	17.5	20.3

preparation. In contrast, the liquid phenolic resin was converted into a cast film when pre-heated at 70 °C for 60 min, eliminating air and preventing combustion. Experimental yields obtained for the hybrid charcoal surpassed those calculated, assuming that each component could be carbonized independently. In addition, the difference between measurements and calculations rose with increasing phenolic resin content. The radicals derived by thermal decomposition seem to have interacted and promoted the polycondensation reaction, especially at the interface of two components, suggesting that a co-carbonization effect occurred in the case of hybrid charcoal [17,18].

3.2. Graphitization of phenolic resin-modified feathers

The composition of the starting material and the chemical structure of each component affect the carbon structure and charcoal yield. Carbon is usually classified into graphitizing and non-graphitizing types. The graphitizing property can be estimated by measuring the graphitization degree P1 [12–14,19], defined as the probability of finding a carbon layer pair that achieves a graphitic ordering in the solid state. In fact, carbonaceous materials with large P1 tend to be transformed into a charcoal with a graphite-like structure. In the case of pure graphite, P1 equals one. Such a densely packed structure, however, is not suitable for the production of AC because the diffusion of activation gases, such as water vapor or CO_2, into carbonaceous materials is inhibited and only an AC displaying limited adsorption-capacity forms. Therefore, the graphitization degree of the starting material should be maintained within a certain range to generate a charcoal suitable for AC applications.

To estimate the graphitizing property of hybrid charcoal, a charcoal produced at 800 °C was heat-treated at 3000 °C, and its P1 index was measured using WAXD (see Table 3). Compared with the indexes of the graphitizing carbons, i.e., TRY (L-tryptophan) and ACE (acenaphthylene) [20], the hybrid charcoal samples, F4Ph1 and F1Ph1, showed fairly small P1 values. However, the P1 index rose with increasing phenolic resin content, from 20% in F4Ph1 to 50% in F1Ph1, which is probably related to the co-carbonization effect observed in the preparation of hybrid charcoal.

The graphitizing ability, or graphitizability, depends on small basic structural units (i.e., crystallites) and the preferred parallel orientation of the structural units during the carbonization process. If the width (La) and the thickness (Lc) of these crystallites are too small, the graphitization becomes impossible [19]. In the previous paper [21] on phenol

formaldehyde resin-based carbon fibers, the increase in Lc resulted in no more than 12 nm at 2040 °C. Thus, phenolic resin should be classified as a non-graphitizing material. The crystallite sizes of the hybrid charcoal after the heat treatment at 3000 °C are listed in Table 3. The sizes are scattered in a range of no more than 12 nm. However, phenolic resin could raise Lc to the level of the hybrid charcoal by ca. 1000 °C lower temperature side. It seems that the graphitizability of phenolic resin is slightly superior to that of feathers. Therefore, the hybridization of phenolic resin can modify the graphitizing property of feathers to some extent.

Compared with graphitizing carbons, it seems that both La and Lc are too small for hybrid charcoals to provide them with graphitizability (see Table 3). Therefore, the hybrid charcoal should be classified as a non-graphitizing carbon.

Fig. 3 shows the WAXD curves of the hybrid charcoals after heat treatment at 3000 °C. Both curves for the 002 diffraction peak became asymmetric, which is a feature typical of non-graphitizing carbon. Moreover, the 002 diffraction peak of F4Ph1 was split, indicating the generation of two types of carbon crystals with different d-spacing.

Overall, the increase in phenolic resin content effectively modifies the graphitizability of feather keratin to some extent through the co-carbonization reaction.
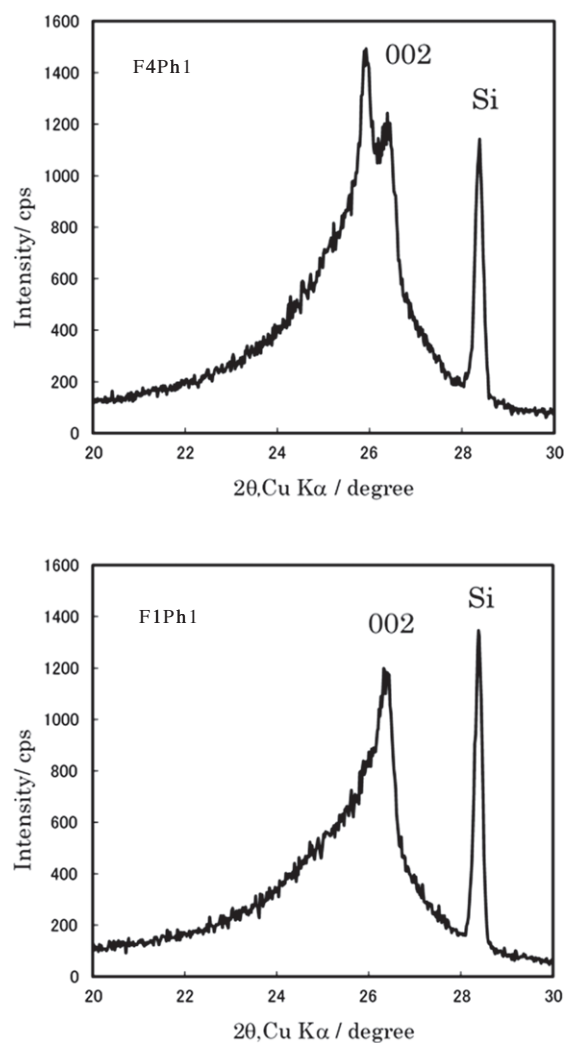
Table 3
Crystallite structure of the carbons produced at 3000 °C.

Sample code	F4Ph1	F1Ph1	TRY [20]	ACE [20]	Pr1S4	CEL [23]
P1	<0.1	0.10	0.57	0.53	0.56	0.13
d_{002}(002), nm	0.3399	0.3374	0.3371	0.3461	0.3377	0.3393
d_{002}(004), nm	0.3426	0.3367	0.3369	0.3361	0.3374	0.3388
d_{002}(006), nm	–	–	0.3370	0.3362	0.3378	–
Lc(002), nm	9	12	47	134	41	14
Lc(004), nm	11	10	28	78	20	8
Lc(006), nm	–	–	15	41	10	–
La(110), nm	3	3	69	161	80	11
σ_c, nm	–	0.007	0.005	0.003	0.006	0.010
L_0, nm	–	12	45	138	44	15

Fig. 3. WAXD curves of hybrid charcoals after heat treatment at 3000 °C. The starting material consists of feathers and the phenolic resin at a mass ratio of 4:1 (F4Ph1, top) or 1:1 (F1Ph1, bottom).

3.3. Co-carbonization reaction of L-proline derivatives

As far as the carbonization behavior of amino acids is concerned, only L-tryptophan has shown a good graphitizing ability despite its absence in feathers [20]. However, we have found peculiar graphitizing behaviors for the binary mixed system consisting of L-proline and natural shellac resin (major constituent: shellolic acid, jalaric acid [22]). L-Proline is one of the major amino acids constructing the backbone of keratin in feathers, and its content is 15.4 wt.% [7].

To clarify the graphitizing behavior of L-proline and natural shellac resin system, a mixture of L-proline and natural shellac resin at a mass ratio of 1:4 (coded as Pr1S4, see Table 1) was carbonized at 800 °C, and then the heat treatment at 3000 °C was conducted. P1 and other crystal parameters measured after the treatment are shown in Table 3. The carbon yield of Pr1S4 at 800 °C was below 10%. However, without the hybridization of L-proline to natural shellac resin, the carbon yield of each component decreased to zero. The P1 value of Pr1S4 was comparable to those of other graphitizing carbons, suggesting that the radicals derived from each component by thermal decomposition have interacted and that a reaction intermediate with a graphitizing structure has been generated. At present, it is difficult to estimate the chemical structure of the reaction intermediate precisely. However, we believe that the reaction intermediate most likely contains a radical structure derived from L-tryptophan residue because the structural parameters measured for the Pr1S4 carbon are similar to those for TRY (see Table 3). Further investigations are needed and will be reported elsewhere.

For the carbonization system containing feathers and phenolic resin, the hybridization could also effectively increase the yield of charcoal. It seems that the co-carbonization reactions induced by L-proline derivatives were affected similarly.

3.4. Production of AC from phenolic resin-modified feathers

Cellulosic compounds are often used as starting materials in the production of AC destined for drinking water and/or wastewater control systems. These materials are classified as non-graphitizing carbon precursors. Therefore, cellulosic compounds extracted from reed grass (CEL) have shown much smaller P1 and crystal parameters than graphitizing carbons (see Table 3) [23]. Non-graphitizing carbons will tend to transform into a charcoal that is rich in amorphous carbon domains and into which activation gases, such as water vapor or CO_2, can diffuse easily. Hence, non-graphitizing carbons are highly attractive for AC production to ensure an effective reaction with activation gases, leading to porous structures with large adsorption capacity. AC materials with BET surface areas exceeding 1000 m^2/g could be easily produced from woody biomass [1,3]. However, if the P1 of the starting material is too small, its liquefaction will occur before carbonization, and therefore the original structure, e.g., the cell structure of woody biomass or the hollow fibrous structure of feathers, will be destroyed.

When structural parameters of the hybrid charcoals were compared with those of CEL (see Table 3), their crystallite sizes were scattered within a similar range although there were slight differences in the P1 value. The F1Ph1 hybrid charcoal prepared at 800 °C was activated

with water vapor, and its surface properties were measured (see Table 4). The BET surface area and the iodine-adsorption capacity for the F1Ph1-based AC were 1.7-1.8 times greater than those of the feather-based AC at the same activation level (approximately 60%). The yield of AC was defined as the relative mass of AC versus the mass of charcoal before activation. However, the BET surface area did not reach the 1000 m^2/g level. If the activation were made deeper, the BET surface area might increase to that level, sacrificing the yield of AC. In contrast, partial liquefaction most likely occurred, leading to the destruction of the hollow fibrous structure of feathers and lowering the reaction surface necessary for the activation process. When we focus on the mean pore diameter determining the application field of AC, it is seen that the value of the F1Ph1-based AC is comparable to that of woody biomass-based AC [1, 3], implying that F1Ph1-based AC can be used to eliminate pollutants from drinking water and wastewater.

4. Conclusions

To expand the utilization of feather waste, the production of AC was attempted. The water-soluble phenolic resin was hybridized to prevent the liquefaction of feathers and to control the graphitization degree of charcoal. The hybridization could effectively increase the yield of charcoal through the co-carbonization effect between feathers and phenolic resin, preventing liquefaction and promoting carbonization. In addition, the hybridization could increase the graphitization degree of the charcoal to a range suitable for the production of AC. The BET surface area and the iodine-adsorption capacity of hybrid charcoal-based AC were 1.7-1.8 times greater than those of the feather-based AC at a similar activation level.

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Table 4
Yields and surface characteristics of ACs.

Sample code	F	F1Ph1
Yield, %		
Carbonization at 800 °C	15.6	31.0
Activation at 900 °C	64.0	59.8
BET area	388	706
BET volume	0.18	0.32
Mean pore diameter	1.83	1.79
Iodine-adsorption capacity, mg/g	320	550

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